

Running spectra at variable temperature.

The temperature of the sample is regulated by a resistor which heats the VT gas flowing from the bottom of the probe to the top. A thermocouple placed at the top measures the temperature (Figure 2). Make sure on the magnet leg that the VT gas is flowing at a rate of 10-30 l/min. Each probe has its own temperature range, which we wrote on the probe (Figure 1).

First decide on the temperature that you want to use. It should be in the liquid range of the solvent, but ca. 5-10 °C away from the melting or boiling point. Also, do not exceed the temperature range of the probe.

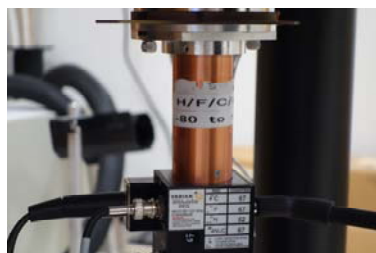


Figure 1.

If cooling below 0 °C switch to nitrogen gas from the valve on the wall. The FTS unit on the VT line will cool to about -60 °C for a VT flow of 30 l/min. You will have to turn it on. To go to lower temperature, ask us to switch the lines to the liquid nitrogen bucket.

For experiments at one temperature, go to Start – Spin/Temp panel and check 'Control temperature from this panel only'. Type in the desired temperature value in the current temperature input box and click regulate temperature. Check that the green VT light next to the temperature display on the interface on top of the console is blinking. It will become steady when the temperature is regulated.

When the temperature has reached the desired value, lock and shim; shimming depends on the temperature.

For 2D work, or if sensitivity is an issue, tune the probe; tuning also depends on temperature.

To acquire spectra at different temperatures in an experiment, make sure that in the Start – Spin/Temp panel the 'Control temperature from this panel only' is turned off. Set up an array of the parameter temp in Acquire – Arrays. Allow some time for the equilibration of the temperature: pad=300 will be 5 minutes. To equilibrate the temperature within 0.1 °C for a 5 °C jump it takes 20 minutes. Set it to shim on automation at each temperature: type wshim='f' , then you can start the acquisition.

Remember to bring back the temperature to 25 °C, turn the VT to air, turn off the FTS and retune the probe, if you changed the tuning.

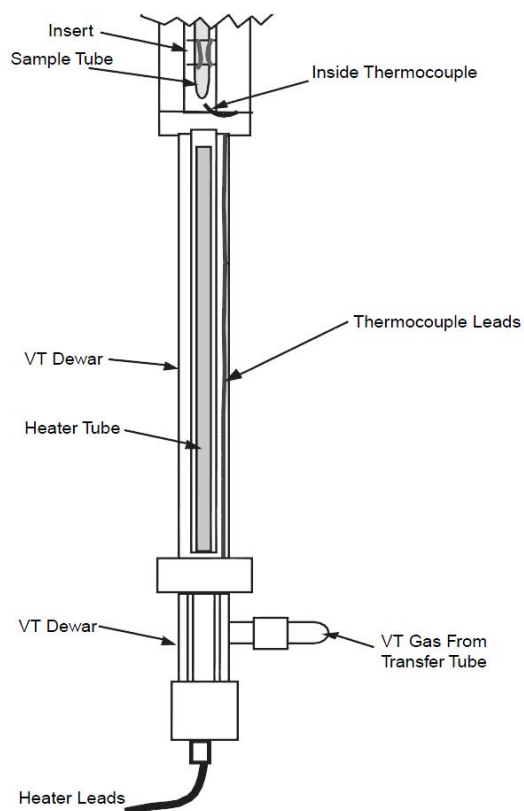


Figure 2. Variable Temperature Probe